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(54) Compositions containing fat-soluble substances in a carbohydrate matrix

(57) The invention relates to compositions containing a fat soluble substance in a glassy carbohydrate matrix comprising maltose or a mixture of low-molecular weight carbohydrates, and, optionally, a high-molecular weight carbohydrate. The compositions can be used for multivitamin tablets, hard gelatin capsules, dry food and feed compositions and for enriching sugar.

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Description

[0001] The present invention relates to compositions comprising fat soluble substances in a glassy carbohydrate matrix, to a process for their manufacture and to their use to enrich food and feed.

[0002] Water soluble compositions of fat soluble vitamins play an important role in the field of human and animal nutrition. Such compositions are usually marketed in the form of emulsions or dry powders. It is a common feature in such compositions that the fat soluble vitamins are usually protected with a matrix component, e.g. a gelatin matrix.

[0003] Stable vitamin compositions have also been conventionally obtained by a method wherein the vitamins are encapsulated in matrixes in the form of a powder. Products on the market are e.g. vitamin A palmitate encapsulated in a CAPSUL® matrix, available under the name Vitamin A Palmitate 250 SD by F. Hoffmann-La Roche AG and vitamin A palmitate encapsulated in a gelatin matrix, available under the name Vitamin A Palmitate 250 CWS by F. Hoffmann-La Roche AG.

[0004] All the products on the market are sensitive to air, heat, light and humidity. Thus, there is a constant need to improve the stability under regular storage conditions. Accordingly, the problem addressed by the present invention was to find compositions comprising fat soluble substances showing an improved storage stability.

[0005] It has now been found that the stability can be improved by encapsulating one or more fat soluble substances in a glassy low-molecular weight carbohydrate matrix.

[0006] Thus, the invention relates to a composition comprising in percents by weight based on the total weight of the composition

from about 1 wt% to about 40 wt% of a fat soluble substance encapsulated in a carbohydrate matrix composed of maltose or maltose syrup, or a mixture of low-molecular weight carbohydrates, optionally in combination with a high-molecular weight carbohydrate; from about 0.1 wt% to about 30 wt% of an emulsifier; and, optionally, from about 0.1 wt% to about 15 wt% of an antioxidant.

[0007] The fat soluble substances include fat soluble vitamins selected from the group consisting of vitamins A, D and K and derivatives thereof; carotenoids such as e.g. beta-carotene, astaxanthin, apocarotenal, canthaxanthin, apoester, citranaxanthin and zeaxanthin; polyunsaturated fatty acids as well as mixtures thereof. Particularly interesting products contain fat soluble vitamins, preferably vitamin A and its derivatives, especially vitamin A acetate or vitamin A palmitate. If the composition comprises a fat soluble vitamin it is advantageous to add an antioxidant. Thus, a preferred composition comprises in percents by weight based on the total weight of the composition from about 1 wt% to about 40 wt% of a fat soluble vitamin encapsulated in a carbohydrate matrix composed of maltose or maltose syrup, or a mixture of low-molecular weight carbohydrates, optionally in combination with a high-molecular weight carbohydrate; from about 0.1 wt% to about 30 wt% of an emulsifier; and from about 0.1 wt% to about 15 wt% of an antioxidant.

[0008] Preferred examples of polyunsaturated fatty acids are selected from the group consisting of arachidonic acid (AA), docosahexaenic acid (DHA) or eicosapentaenic acid (EPA).

[0009] Low-molecular weight carbohydrates include mono- and di-saccharides such as e.g. fructose, glucose, glucose syrup, sucrose, lactose, dextrose, maltose, high-maltose solid (syrup), xylose, arabinose, ribose and sugar alcohols. Especially preferred are fructose, glucose, glucose syrup, maltose and sucrose. Maltose can be used also in the form of high-maltose solid (syrup) which contains over 50 wt% of maltose.

[0010] The low-molecular weight carbohydrates are used at a level of about 30 wt% to about 95 wt%, preferably of about 50 wt% to about 85 wt%, more preferably about 70 wt%.

[0011] High-molecular weight carbohydrates include e.g. maltodextrin, which is used at a level of 0 wt% to about 50 wt%, preferably about 10 wt% to about 40 wt%, more preferably about 30 wt%.

[0012] Maltodextrin can be obtained from Grain Processing Corp. under the trade name MALTRIN.

[0013] Suitable emulsifiers are polyoxyethylene-sorbitan-fatty acid esters; e.g. mono- and tri-lauryl, palmityl, stearyl and oleyl esters; especially those available under the tradename TWEEN (for example TWEEN 80, TWEEN 60, TWEEN 40, TWEEN 20) from ICI, chemically modified starch obtainable from National Starch & Chemical Company under the tradename CAPSUL and HI-CAP, and ascorbyl palmitate.

[0014] Suitable antioxidants are selected from the group consisting of sodium ascorbate, ascorbyl palmitate, dl-α-tocopherol, mixed tocopherols, lecithin, butylated hydroxy toluene (BHT), butylated hydroxy anisole (BHA) and mixtures thereof. Preferred are sodium ascorbate, ascorbyl palmitate, dl-α-tocopherol, mixed tocopherols and lecithin.

[0015] The antioxidants can be added either to the aqueous phase and/or to the lipid phases. Sodium ascorbate is preferably added to the aqueous phase. Ascorbyl palmitate and/or dl-α-tocopherol are preferably added to the lipid phase.

[0016] The compositions in accordance with the invention can be manufactured, in principle, by preparing an oil in water emulsion containing from about 1 wt% to about 40 wt% of a fat soluble substance; from about 30 wt% to about 85 wt% of maltose or a mixture of low-molecular weight carbohydrates optionally in combination with 0 wt% to about 50 wt% of a high-molecular weight carbohydrate; from about 0.1 wt% to about 30 wt% of an emulsifier; and, optionally, from about 0.1wt% to about 15wt% of an antioxidant; and, if desired, converting this emulsion into a dry powder.

[0017] It is self evident that the total amount of the ingredients is not beyond 100wt%.

[0018] Generally the low-molecular weight carbohydrates optionally in combination with high-molecular weight carbohydrates are first dissolved in water. It is advantageous to carry out this process step at a temperature in the range of about 20°C to about 90°C, preferably about 40°C to about 75°C. Then the antioxidant and the emulsifier are added. The so called carbohydrate matrix is obtained in this manner. Then, the fat soluble substance or a mixture of several such substances is mixed with an antioxidant, if desired, and the resulting mixture is gradually added to the aqueous phase while the mixture is homogenized with a mixer to form an oil in water emulsion. The procedure can be carried out readily at temperatures of about room temperature to about 80°C, preferably at about 30°C to about 50°C, more preferably about 40°C.

[0019] The conversion of a thus-manufactured emulsion into a dry powder can be effected by methods known in the art e.g. by spray drying.

[0020] The compositions in accordance with the invention show an excellent stability at temperatures up to 35 °C and show a better stability under humid condition. The use of a low-molecular weight sugar mixture prevents sugar crystallization from the sugar glass matrix and thus, the stability of the fat soluble substance, particularly the stability of fat soluble vitamins under humid stress conditions is improved.

[0021] The compositions in accordance with the invention can be used for multivitamin tablets, hard gelatin capsules and dry food and feed compositions.

[0022] Furthermore, the composition can be mixed directly without using any adhesive with sugar, e.g. with sucrose. This is an essential advantage as the prior art products Vitamin A Palmitate 250 SD or Vitamin A Palmitate 250 CWS require the use of oil as an adhesive to ensure homogeneity and no segregation.

[0023] To enrich sugar it is advantageous to prepare a premix by mixing sugar and the dry powder of the composition according to the invention in a ratio of about 14 to 1 to about 4 to 1. The sugar crystals are preferably wetted before being added to the dry powder by adding a small amount of a saturated sucrose solution or of water. To reduce its hygroscopicity it is advantageous to coat the premix with an anticaking agent such as silicic acid or with silicate by simply shaking the premix with the anticaking agent. The anticaking agent is added in an amount of about 0.2 wt% to about 2 wt%.

[0024] The present invention is illustrated by the following examples:

Example 1

[0025] Starch sodium octenyl succinate (84.0 g; CAPSUL from National Starch & Chemical, Bridgewater, NJ) was dissolved in water (402 g) and heated to 65 °C. Sucrose (461.5 g) and maltodextrin (243.1 g; MALTRIN M100; Grain Processing Corp., Muscatine, Iowa) were then dissolved in the starch solution and the temperature was held at about 65 °C. Sodium ascorbate (15 g) was then added to the sucrose solution and the solution was held at 40 °C. Water lost due to evaporation was made up before homogenization with the lipid phase. A mixture of vitamin A palmitate (179.6 g), dl-α-tocopherol (15.75 g) and ascorbyl palmitate (15.75 g) was stirred and heated to 40 °C and then stirred at said temperature for about 15 minutes. The lipid phase mixture (201 g) was then gradually added to the sucrose solution and homogenized under nitrogen with a homogenizer (Gifford-Wood homogenizer) to yield an emulsion having a particle size of approximately 0.2-1.5 microns. The viscosity of the emulsion was adjusted with additional water, if necessary. The emulsion was spray-dried (Niro Atomizer, Copenhagen, Denmark) to give a powder.

Example 2

[0026] Starch sodium octenyl succinate (84.0 g; CAPSUL from National Starch & Chemical, Bridgewater, NJ) was dissolved in water (374 g) and heated to 65 °C. Maltose (368.2 g) and maltodextrin (364.7 g; MALTRIN M100; Grain Processing Corp., Muscatine, Iowa) were dissolved in the starch solution and the temperature was held at about 65 °C. Sodium ascorbate (15 g) was then added to the sucrose solution and the solution was held at 40 °C. Water lost due to evaporation was made up before homogenization with the lipid phase. A mixture of vitamin A palmitate (179.6 g), dl-α-tocopherol (15.75 g) and ascorbyl palmitate (15.75 g) was stirred and heated to 40 °C and then stirred at said temperature for about 15 minutes. The lipid phase mixture (201 g) was then gradually added to the sucrose solution and homogenized under nitrogen with a homogenizer (Gifford-Wood homogenizer) to yield an emulsion having a particle size of approximately 0.2-1.5 microns. The viscosity of the emulsion was adjusted with additional water, if necessary. The emulsion was spray-dried (Niro Atomizer, Copenhagen, Denmark) to give a powder.

Example 3

[0027] Starch sodium octenyl succinate (84.0 g; CAPSUL from National Starch & Chemical, Bridgewater, NJ) was dissolved in water (366 g) and heated to 65 °C. Sucrose (69.2 g), Glucose syrup (88 g), maltose (73.6 g), glucose (76.0

g), fructose (69.2 g) and maltodextrin (364.7 g; MALTRIN M100; Grain Processing Corp., Muscatine, Iowa) were dissolved in the starch solution and the temperature was held at about 65 °C. Sodium ascorbate (15 g) was then added to the sucrose solution and the solution was held at 40 °C. Water lost due to evaporation was made up before homogenization with the lipid phase. A mixture of vitamin A palmitate (179.6 g), dl-α-tocopherol (15.75 g) and ascorbyl palmitate (15.75 g) was stirred and heated to 40 °C and then stirred at said temperature for about 15 minutes. The lipid phase mixture (201 g) was then gradually added to the sucrose solution and homogenized under nitrogen with a homogenizer (Gifford-Wood homogenizer) to yield an emulsion having a particle size of approximately 0.2-1.5 microns. The viscosity of the emulsion was adjusted with additional water, if necessary. The emulsion was spray-dried (Niro Atomizer, Copenhagen, Denmark) to give a powder.

Example 4

[0028] Starch sodium octenyl succinate (84.0 g; CAPSUL from National Starch & Chemical, Bridgewater, NJ) was dissolved in water (377 g) and heated to 65 °C. Sucrose (115 g), maltose (122.3 g), glucose (126.2 g) and maltodextrin (364.7 g; MALTRIN M100; Grain Processing Corp., Muscatine, Iowa) were dissolved in the starch solution and the temperature was held at about 65 °C. Sodium ascorbate (15 g) was then added to the sucrose solution and the solution was held at 40 °C. Water lost due to evaporation was made up before homogenization with the lipid phase. A mixture of vitamin A palmitate (179.6 g), dl-α-tocopherol (15.75 g) and ascorbyl palmitate (15.75 g) was stirred and heated to 40 °C and then stirred at said temperature for about 15 minutes. The lipid phase mixture (201 g) was then gradually added to the sucrose solution and homogenized under nitrogen with a homogenizer (Gifford-Wood homogenizer) to yield an emulsion having a particle size of approximately 0.2-1.5 microns. The viscosity of the emulsion was adjusted with additional water, if necessary. The emulsion was spray-dried (Niro Atomizer, Copenhagen, Denmark) to give a powder.

Example 5

(Stability Evaluation)

[0029] Each sample prepared as described in Examples 1-4 was mixed sucrose (cane sugar) in a ratio of 1 to 4. The mixture was then stored in sealed polyethylene bags at 37 °C/75% relative humidity for vitamin A stability evaluation. The % Vitamin A palmitate retentions at various time intervals are shown in the following Table 1. It shows that the samples prepared with maltose only (Example 2) or with a mixture of low molecular weight carbohydrates (Example 3 and 4) according to the invention have over-all good stability, whereas the sample prepared with sucrose only (Example 1) shows a sudden loss of vitamin A after 1.5-month storage, which significantly reduces the shelf-life of the product.

Table 1

	Example 1	Example 2	Example 3	Example 4
Sucrose	46.15	0	6.92	11.5
Maltose	0	34.61	6.92	11.5
Glucose syrup	0	0	6.92	0
Glucose	0	0	6.92	11.5
Fructose	0	0	6.92	0
Maltrin M100	23.07	34.61	34.61	34.61
Capsul	7.69	7.69	7.69	7.69
Vitamin A Palmitate	17.1	17.1	17.1	17.1
Sodium Ascorbate	1.5	1.5	1.5	1.5
α-DL-Tocopherol	1.5	1.5	1.5	1.5
Ascorbyl Palmitate	1.5	1.5	1.5	1.5
Water	(1.5)	(1.5)	(1.5)	(1.5)
Total	100	100	100	100
% Vitamin A Retention at 37 °C/75% RH				
Initial	100	100	100	100
0.5 months	99.1	89.8	91.2	94.2
1.0 months	104	99.0	96.6	105
1.5 months	100	92.7	94.1	94.0
2.0 months	0	92.9	99.2	87.7
2.5 months	0	86.7	55.9	52.8
3.0 months	0	73.9	54.2	47.8

Example 6

[0030] Preparation of a sugar premix containing silicic acid.

[0031] Starch sodium octenyl succinate (84 g; CAPSUL from National Starch & Chemical, Bridgewater, NJ) was dissolved in water (379 g). Sucrose (115.4 g), maltose (120.2 g), glucose (126.6 g), fructose (122.7 g) and maltodextrin (243.1 g; MALTRIN M100; Grain Processing Corp., Muscatine, Iowa) were dissolved in the starch solution and the temperature was raised to about 65 °C. Sodium ascorbate (15 g) was then added to the sucrose solution and the solution was held at 40°C. Water lost due to evaporation was made up before homogenization with the lipid phase. A mixture of vitamin A palmitate (188.1 g), dl-α-tocopherol (16.5 g) and ascorbyl palmitate (16.5 g) was stirred and heated to 40 °C and then stirred at said temperature for about 15 minutes. The lipid phase mixture (201 g) was then gradually added to the sucrose solution and homogenized under nitrogen with a homogenizer (Gifford-Wood homogenizer) to yield an emulsion having a particle size of approximately 0.2-1.5 microns. The viscosity of the emulsion was adjusted with additional water, if necessary, and the emulsion was spray-dried (Niro Atomizer, Copenhagen, Denmark) to give a powder.

[0032] A saturated sucrose solution was prepared by dissolving sucrose (100 g) in water (50 g). The saturated sucrose solution (15 g) was added to sucrose crystals (600 g) and then mixed until the crystals were evenly wetted. The wet crystals (615 g) were gradually added to the spray-dried powder (150 g) prepared as described above while the mixture was gently mixed. The mixture was dried in a desiccator over the weekend and then equilibrated in a desiccator containing calcium chloride (about 30% relative humidity). The mixture (490 g) was then coated with silicic acid (7.35 g) by shaking with silicic acid to reduce its hygroscopicity.

Claims

1. A composition comprising in percents by weight based on the total weight of the composition

   from about 1 wt% to about 40 wt% of a fat soluble substance encapsulated in a carbohydrate matrix composed of maltose or maltose syrup, or a mixture of low-molecular weight carbohydrates, optionally in combination with a high-molecular weight carbohydrate;

   from about 0.1 wt% to about 30 wt% of an emulsifier; and, optionally,

   from about 0.1 wt% to about 15 wt% of an antioxidant.

2. A composition according to claim 1, wherein the carbohydrate matrix is composed of a mixture of low-molecular weight carbohydrates in combination with a high-molecular weight carbohydrate.

3. A composition according to claims 1 or 2, wherein the low-molecular weight carbohydrates are selected from the group consisting of fructose, glucose, glucose syrup, sucrose, lactose, dextrose, maltose, high-maltose solid (syrup), xylose, arabinose, ribose and sugar alcohols.

4. A composition according to claims 1 or 2, wherein the low-molecular weight carbohydrates are selected from the group consisting of fructose, glucose, glucose syrup, maltose and sucrose.

5. A composition according to claim 1, wherein the carbohydrate matrix is composed of maltose or high-maltose solid (syrup) containing over 50 wt% of maltose, in combination with a high-molecular weight carbohydrate.

6. A composition according to any one of claims 1 to 5, wherein the high-molecular weight carbohydrate is maltodextrin.

7. A composition according to claim 1 to 6, wherein the fat soluble substance is a fat soluble vitamin selected from the group consisting of vitamins A, E, D and K and derivatives thereof, a carotenoid, a polyunsaturated fatty acid as well as mixtures thereof.

8. A composition according to of claim 7, wherein the vitamin A derivative is vitamin A acetate or vitamin A palmitate.

9. A composition according to claim 7, wherein the carotenoid is selected from the group consisting of beta-carotene, astaxanthin, apocarotenal, canthaxanthin, apoester, citranaxanthin and zeaxanthin.

10. A composition according to claim 7, wherein the polyunsaturated fatty acid is selected from the group consisting of arachidonic acid (AA), docosahexaenic acid (DHA) or eicosapentaenic acid (EPA).

11. A composition according to any one of claims 1 to 10, comprising from about 30 wt% to about 95 wt%, preferably from about 50 wt% to about 85 wt%, more preferably about 70 wt% of low-molecular weight carbohydrate.

12. A composition according to any one of claims 1 to 11, comprising from 0 wt% to about 50 wt%, preferably from about 10 wt% to about 40 wt%, more preferably about 30 wt% of high-molecular weight carbohydrate.

13. A composition according to any one of claims 1 to 12, wherein the emulsifier is a polyoxyethylene-sorbitan-fatty acid ester, a chemically modified starch or ascorbyl palmitate.

14. A composition according to any one of claims 1 to 13, wherein the antioxidant is selected from the group consisting of sodium ascorbate, ascorbyl palmitate, dl-α-tocopherol, mixed tocopherols, lecithin and mixtures thereof.

15. A premix for enriching food comprising a composition according to any one of claims 1-14, and sugar.

16. A process for preparing a composition according to any one of claims 1-14, which process comprises preparing an oil-in-water emulsion containing from about 1 wt% to about 40 wt% of a fat soluble substance; from about 30 wt% to about 85 wt% of maltose or a mixture of low-molecular weight carbohydrates optionally in combination with 0 wt% to about 50 wt% of a high-molecular weight carbohydrate; from about 0.1 wt% to about 30 wt% of an emulsi-

fier; and, optionally, from about 0.1wt% to about 15wt% of an antioxidant; and, if desired, converting this emulsion into a dry powder.

17. The use of the compositions according to any one of claims 1 to 14 for multivitamin tablets, hard gelatin capsules and dry food and feed compositions.

18. Process for preparing a premix which process comprises mixing sugar and the composition according to any one of claims 1 to 12 in form of a dry powder in a ratio of about 14 to 1 to about 4 to 1.

19. Process according to claim 18, whereby the sugar is wetted before being added to the dry powder by adding a small amount of a saturated sucrose solution or of water.

20. Process according to claim 18 or 19, whereby an anticaking agent is added, preferably in an amount of about 0.2 wt% to about 2 wt%.

21. Process according to claim 20, wherein the anticaking agent is silicic acid or silicate.

22. The novel compositions, premixes, processes and use substantially as described hereinbefore, especially with reference to the Examples.

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EUROPEAN SEARCH REPORT

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TECHNICAL FIELDS SEARCHED (Int.Cl.7)
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The present search report has been drawn up for all claims

Place of search	Date of completion of the search	Examiner
MUNICH	23 May 2001	Vernier, F

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